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# THE POTENTIAL OF MICROWAVE RADIATION FOR PROCESSING MARTIAN SOIL


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## Introduction

For extended or permanent stay operations on Mars, in situ resource utilization is necessary due to the high cost of space transportation. Using the soil and atmosphere of Mars for the extraction of important materials is essential. This work investigates the potential use of microwave radiation on a Martian soil analog to aid in beneficiation. The work is both exploratory and experimental. Results of experiments are presented and discussed.

## Literature Review

Rao [5] reports the results of research using microwave heating of bauxite and hematite for the removal of iron. He observed formation of agglomerates, formation of metallic iron (longer heat – more iron), an increase in magnetic material from 0% to 29.5% by weight in the case of hematite, and from 22% to 75% in the case of bauxite (with coke as a reducing agent in both cases). The objective was to rid the ore of the contaminant iron. Rao looked for the chemical reaction as shown in Formula 1.

$$2Fe_2O_3 + 3C = 3CO_2 + 4Fe \qquad (1)$$

Kingman and Rowson of Birmingham University (UK) [3] report results of comparing microwave heating and conventional muffle furnace: "The results are very similar for both forms of heat treatment, showing that microwave energy produces the same overall structure changes, but in a more rapid time period." Similarly, Kingman, et al. [2] report the effect of microwave radiation upon a massive Norwegian ilmenite: "Short exposure to microwave radiation has been demonstrated to cause fractures within the ore matrix. Increased exposure to microwave radiation is shown to cause localised sample melting. The microwave treated samples have subsequently undergone a multi-stage magnetic separation process which produced concentrates of significantly higher grade and also better recovery of valuable mineral, when compared to those that are not treated."

Some authors such as Reid (http://home.c2i.net/metaphor/mvpage.html) have looked at melting metals in a domestic microwave oven. Meek, et al. [4], describe potential benefits of using microwave radiation on lunar soils as energy efficient and discuss how selected frequencies may couple with specific bonds. They also experimented by melting ore and developing bricks. Tucker, et al., [6] describe a method of hydrogen recovery from extraterrestrial materials: "The use of microwave energy would offer a new, efficient method of heating lunar materials. Not only could extraterrestrial materials be heated with less energy than with conventional methods, but the heating would be accomplished in a controlled manner and in much less time than with a method such as solar heating."

Most of the work reviewed for this effort looks at the use of microwave energy in making bricks rather than beneficiating and extracting elements out of Lunar or Martian soils. It appears very little experimentation has been accomplished in beneficiation for the extraction of metals and other useful elements with microwave energy. No references were found that used microwave energy on JSC Mars-1 Martian soil simulant.

## Objective

The objective of this research is to microwave a Martian soil Simulant, JSC Mars-1 [1], in a conventional microwave oven with graphite (carbon) as a reducing agent. This should result in some of the iron oxide reacting as indicated in Formula 1, thereby, creating Fe that should be more easily removed by magnetic separation.

**Estimating Carbon Content Required**

For the reaction as depicted in Formula 1, each gram of Mars-1 requires about 0.0169 g of C, or 1.7% by weight. This may be theoretically accurate. However, there is a proximity issue. The carbon must be close to the iron oxide to react and the location of the iron oxide in the sample is not spatially homogenous. Therefore, it may take more than the amount indicated.

**Procedure**

A "hot spot" was located within the microwave (Sharp R-230BK, 700 W) using heat sensitive printer paper. All tests were run with an alumina thimble crucible placed on an alumina plate over the hot spot for 8 minutes. Preliminary efforts revealed significant blowout of material from the crucible anywhere from 10 to 50 seconds after starting the microwave. Shorter time to blowout was noted for higher carbon concentrations. It was initially believed this was due to the presence of water. However, even with samples dried at 150° C for 24 hours, blowout still occurred. This was assumed to be due to adsorbed water that doesn't evaporate until temperatures reach 350° C and is completely gone at 500°C. A sample was prepared and subjected to 500+° C for 3 hours. The sample was immediately heated in the microwave and blowout still occurred. This suggests that the reaction is not due to the presence of water.

Materials were stored in an oven at 60° C. After running preliminary samples a final group of samples was prepared with the carbon content increased from 2% to 10% in increments of 2%. No reaction was observed for the 2% sample. The sample was cool enough to touch by hand only after a minute or two of cooling. The 4% and 5% samples exhibited glowing red heat and slightly sintered in the center of the crucible. The 8% and 10% samples exhibited bright red to white heating and produced melted samples. Since blowout occurred in all samples, it is impossible to know the carbon content of the remaining portion.

In order to see if the grain size of the carbon had an effect on the blowout, a sample of carbon was crushed with a hammer to produce a coarse grain size. A soil-carbon sample was prepared with a carbon content of 10% coarse graphite. There was no visible reaction when irradiated in the microwave oven. There is obviously a critical relationship between grain size and carbon content. This may be due to smaller spaces between carbon particles in the small grain size portion, thus more easily allowing electrical arcing and resistive heating to occur.

Magnetic separation of the Mars-1 was accomplished using a vertical acrylic tube through a superconducting electromagnet set at 35 kilogauss. Approximately 35% of the sample was retained in the magnetic field within the tube and 65% passed through. Several samples were run on the magnetic and nonmagnetic portions. Visually, there was no difference in the heating of the magnetic portion at 6% carbon and the nonmagnetic portion at 12%. Both samples produced a completely melted bead. The magnetic portion appeared to produce less blowout of material. Time did not allow sample analysis prior to the deadline for this paper.

**Discussion**

Industrial extraction of iron is normally accomplished in a blast furnace where iron ore, rich in iron oxide, is heated to 1200° C to 1300° C with carbon as a reducing agent. The minimum temperature required for the reaction in Formula 1 to take place is about 800° C. Depending upon the type of furnace, carbon may be used both as the reduction agent as well as burned to provide the heat required. When using microwave energy it is difficult to use carbon as a reactant and as a susceptor for microwave energy. The difficulty seems to be that at small concentrations or large grain size no reaction occurs and heating is limited to just over 100° C. At larger concentrations a very vigorous reaction occurs which sinters or melts the sample. From visual observation of color (almost white), the sample appears to reach well over 1000° C. The carbon either does not react at low concentrations or it reacts vigorously at higher concentrations. There seems to be little, if any, middle ground. Controlling heat using this method is likely to be a very difficult problem. Maintaining temperatures for periods of time longer than a few minutes is also difficult because the carbon is consumed. Thus, the original objective of transforming iron oxide into a more magnetically sensitive material in granular form was not accomplished.

**Sample Analysis**

Several melted samples were mounted and microprobe analysis performed. Perhaps the most interesting result is that iron elements seem to precipitate out and form tiny spheres of iron, iron phosphate, and iron silicide. Figure 1 (Left) shows an area of iron micro spheres surrounded by iron depleted glass melt. Figure 1 (Center) shows where iron micro spheres have apparently migrated to the perimeter of the melted bead. Figure 1 (Right) depicts a micro sphere at higher magnification. The darker shaded areas are predominately iron while the lighter shaded areas also include significant amounts of phosphorus. Other metals are sometimes included in the spheres such as titanium. Interestingly, iron phosphate and iron silicide do not occur naturally except in fulgurites (sandy soil melted by lighting). The larger spheres generally appear more at the edges and bottom of melted samples. It seems that the more vigorous the melt, the more spheres are formed and migrate to the edge and bottom of the melt. Perhaps gravitational pull helps the heavy spheres settle toward the bottom.

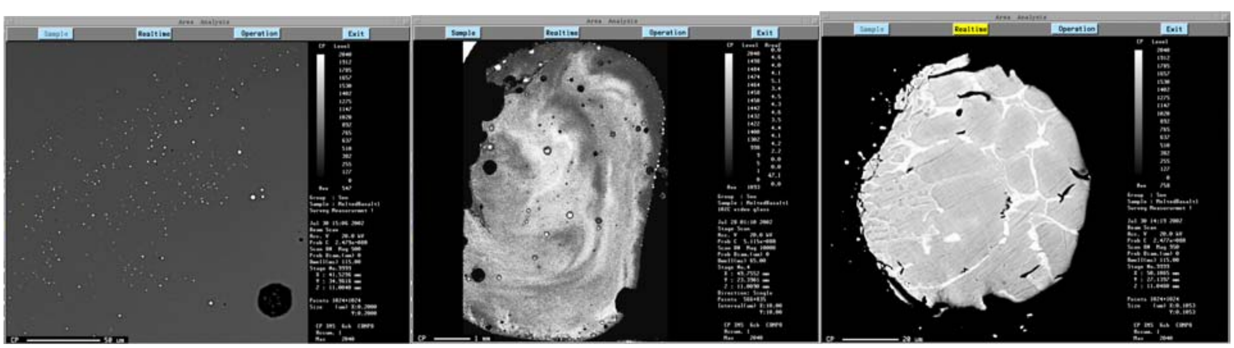

**Figure 1**. Microprobe Photos: Left—iron micro spheres within glass melt; Center—micro spheres at perimeter; Right—close-up view of micro sphere

**Recommended areas for Further Study**

- Heating mechanism: distributed susceptor material within non-susceptor matrix
    - Susceptor material, grain size, spacing, reactivity
    - Does susceptor material provide resistive heating through electrical circuits?
- Microwave radiation variables: frequency, focusing, power
- Controlling heat: producing a melt or heat below melt temperature

- What gas comes off early on?
- Is microwave irradiation an efficient energy delivery method in terrestrial steel making?
- Will iron form and settle to the bottom of the melt the way it does in steel making?
- Will a magnetic field aid in the collection of magnetic spheres (bottom, for example)?
- Will other chemical additives increase yield?

**Conclusion**

While this research is not conclusive, one can make the following comments regarding the potential benefits of microwave radiation for ISRU purposes:

- Microwave radiation with a susceptor material (carbon) will produce intense heat, produce a glassy melt material, and facilitate the formation of metallic micro spheres.
- Microwave equipment is typically low in mass, low in energy consumption, and simple
- Further research is required to determine the degree of purity obtainable whether by single batch operation or successive enrichment procedures.
- A robotic mission design is certainly feasible with existing technology.

This research effort was simple in concept. However, the results turned out to be very complex and created many questions and avenues for further research. The strikingly vigorous reactions obtained were quite unexpected. The concept of a fine-grained, distributed susceptor that may create electrical pathways for resistive heating is interesting per se. The potential of enhancing the yield by adding other chemicals and increasing reclaim efficiency with the use of a magnetic field during processing are also titillating. I wish to acknowledge the assistance of my NASA colleague, Ben Penn and URSA colleagues Laurent Sibille, Subhayu Sen, and Paul Carpenter.

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